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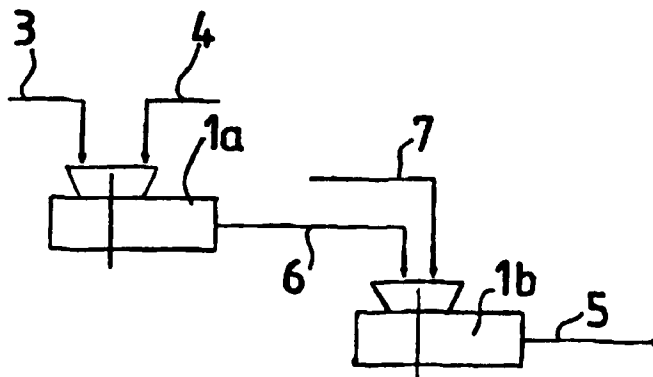
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(54) Title: METHOD AND APPARATUS FOR SLURRYING PULVERIZED MATERIALS INTO A LIQUID

(57) Abstract

The invention relates to a method and an apparatus for preparing slurries of high solids content and high viscosity from liquids and pulverized materials. When preparing such slurries, the mixing of dry solids into the slurry turns difficult as the solids content becomes higher. The invention is based on preparing the slurry in at least two steps through first preparing a slurry with a solids content lower than that of the final product and then mixing into this slurry more solids in at least one step until the desired solids content is attained. Mixing is performed in a pin-mill-type apparatus (1a, 1b) incorporating concentric rings equipped with impact stops which are rotated so that a tangential speed difference is attained between the impact stops.



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Method and apparatus for slurring pulverized materials into a liquid

5 The present invention relates to a method according to the preamble of claim 1 for the preparation of slurries with high solids content and high viscosity from different kinds of pulverized materials and liquids.

10 The invention also concerns an apparatus according to the preamble of claim 11 for implementing the method.

15 Paper industry uses large quantities of various coating materials that are applied on the paper web to achieve desired properties of the sheet. Such coating formulas comprise water slurries of pulverized pigments, fillers and other additives. While similar mixtures are also used in other industries, the prepared quantities generally remain much smaller than in the paper industry. In the paper industry it is important that the solids content of the slurry is high, and correspondingly, the water content low. One reason thereto is that the slurry must occasionally be transported over a longer distance in the paper mill, whereby high water content increases the batch volume of the slurry, and resultingly, the workload in its transportation. A more important reason for aiming at high solids content is that paper coating typically aims at applying a maximum coat weight on the web using one applicator unit only. Moreover, the applied wet coat must be dried prior to further processing of the sheet, whereby the evaporation of excess water consumes abundant energy and is difficult at current web speeds if the web is allowed to wet excessively. With higher web speeds, the drying speed becomes a limiting factor in the construction of high-speed machines requiring a longer path in the dryer, whereby the paper machine becomes longer. Thus, the solids content of the coating affects many aspects of paper machine design and paper manufacture.

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Typically, dispersion into a slurry is carried out in a dissolver-type mixer in which a rotating disk resembling a circular saw blade imposes large shearing forces locally in the slurry causing wetting of the pigment particles and adherence of dispersing agents on the surface of the particles. The coating mix may consist of, e.g., kaolin, talc, titanium oxide or modified forms or combinations thereof. Generally, the slurry is made up of several agents to achieve desired properties of the mixture. A mixer of improved degree of slurring and specific energy efficiency is described in, e.g., FI Pat. No. 91,602. The apparatus disclosed in this publication comprises a pin-mill-type mixer equipped with special-design agitating blades that impose extremely high shearing forces on the mix to be agitated.

Irrespective of what kind of mixing apparatus is used for slurring, the mixing action involves difficulties which become more serious when the viscosity of the mix increases. For liquids of different viscosity, the degree of mixing efficiency can be estimated by means of a correction factor K ($K > 1$) of the mixing time. The correction factor can be determined from formula (1) obtained by experimental methods:

$$K = (\text{Dens}_1 / \text{Dens}_2)^{0.25} \times v^p \quad (1)$$

$p = (0.25 + 0.0025V)$, where

V is the ratio of viscosities of liquids being mixed, and

ratio of densities of liquids is expressed by the ratio in parentheses.

Solving the equation for different values makes it evident that p becomes larger than 1 when $V > 300$, wherein after the mixing becomes rapidly more difficult requiring a longer mixing time if the difference between the

viscosities increases. The equation is given in publication Chem. Engn. World (India) 7, 1972, No. 3, pp. 41-46, Daftary, N.N. Experimental investigations concerning the validity of the equation for viscosity ratios above 100 have not been carried out. While the ratio of densities in the slurring of pulverized solids and liquids is in the order 2.5 - 2.7 and can thus be determined readily, it must be noted, however, that the ratio of densities cannot be defined in the conventional sense when it increases to an extremely high value under certain conditions.

When dry, pulverized material is added in water, a portion of the pulverized material exists agglomerated into large masses which fall as lumps into the liquid. Lumping and agglomeration occurs also when pulverized material accumulates moisture and becomes lumpy in the liquid prior to its complete slurring into liquid. Such lumps first absorb water by capillary forces until all capillary spaces of the lump become filled with water. The internal viscosity of such a lump, that is, the force required for breaking or reshaping the lump becomes extremely high. For instance, if the internal viscosity of a pigment slurry is 500 cP at 55 % solids content of the slurry, the solids content of the lumps is approx. 75 %, whereby their viscosity must be estimated at 100,000 cP. Independently from its actual viscosity value, a lump behaves like a putty. Therefore, slurring of such a material in water is almost impossible in accordance with Eq. 1 due to the high viscosity ratio. This has also been found true in practice.

While a hard putty-like material can be dispersed in water where the viscosities are always low, the putty with the above-described properties is extremely difficult to disperse. However, many materials can be dispersed well if the solids content is kept sufficiently

low. This is because during the mixing action, the turbulent movement of water erodes the dry, surface-wetted lump from its outer surface and rapidly separates the agglomerated particles from each other. Relatively
5 easy mixing of coating slurries results if the dry solids of the slurry are in the range 0 - 40 %. At higher solids, the mixing action and breaking-up of the lumps become more difficult.

10 At 67 - 70 % solids, a typical dissolver mixer slurries, e.g., kaolin or calcined kaolin down to 50 % solids in about 45 min in a batch of 6 - 7 m³. Then, the mixer power input is approx. 250 kW making the specific energy consumption to be at approx. 22 kWh per ton slurry. Not-
15 withstanding the long slurrying time, the raw slurry must be screened to remove unbroken lumps.

The above-cited FI Pat. No. 91,602 describes a partial solution to the slurrying problems. In the mixer dis-
20 closed therein, the shearing forces are made so large as to achieve good breaking-up of even the putty-like lumps. A disadvantage of cited embodiment is in that the shearing forces and the statistical distribution thereof are not necessarily sufficient to provide a homogeneous
25 slurry when the water and the pulverized or ground material are fed directly into the slurrying apparatus. Another problem in such an apparatus is related to consistent dosing of the materials to be mixed, because herein the amounts to be mixed simultaneously are small
30 and the mixing time is short.

The above-described problems can be overcome by virtue of the present invention through a novel application of Eq. 1 which characterizes the mixing operation. When the
35 problematic task is to disperse a high-density, high-viscosity agglomerated lump wetted by the capillary action into a liquid, according to Eq. 1 also that liquid

into which the agglomerated lump is to be dispersed must have a high density combined with a high viscosity.

5 This concept was tested by slurrying a portion of calcined kaolin up to approx. 40 - 45 % solids content in a first step using an apparatus resembling a pin mill. In a second step, into this slurry of already relatively high density and high viscosity was dispersed more kaolin until a 52 % solids slurry was obtained. It was found
10 that this two-step arrangement results in easy preslurrying and final slurrying as the two-step method can avoid large density and viscosity differences in the slurrying action.

15 It is an object of the present invention to achieve a method and apparatus offering superior simplicity over the prior art in the slurrying of pulverized materials into liquids to form a slurry of high solids content, density and viscosity.

20 The goal of the invention is achieved by virtue of performing the preparation of the slurry in at least two steps so that the first step comprises preparing a mixture having a solids content lower than that of the final
25 product, and to this slurry is mixed in at least one subsequent step more solids until the desired solids content of the slurry is attained.

30 According to a preferred embodiment, the slurrying is performed using a slurrying apparatus resembling a pin mill in which the mixture is slurried with a portion of the solids up to such a solids content that corresponds to approx. 70 - 85 % of the desired final solids content and the rest of the solids is slurried into this high-
35 density, high-consistency slurry of already high homogeneity. The second slurrying step is advantageously carried out using again a similar pin mill apparatus.

According to a further preferred embodiment, the pin mill used in the first step has stator vanes and rotor vanes, and the pin mill of the second step has the pins advantageously replaced by lug vanes, and both vane-carrying
5 rotors are arranged to rotate in opposite directions.

According to a still further preferred embodiment, the solids feed to both slurring steps is brought in contact with water only after the solids have first been passed
10 through a similar pin-mill-like apparatus preceding the mixing step with the water. Thus, all above-mentioned solids agglomerations are eliminated as they are disintegrated in the pin mill. This concept is based on the fact that disintegration of agglomerations is easy either
15 a) as entirely dry, or, b) as wet in the dry solids range 0-45 %, while difficulties will arise with dry solids exceeding 45 % and extreme difficulties will be involved in the dry solids range of 55-75 %.

20 As according to observations made by the inventors it is advantageous to prepare a slurry from dry, pulverized material into a ready-made water slurry of solids having a density above that of water, according to the invention at least a portion of the solids is slurried into a
25 slurry. The slurring apparatus comprises advantageously one or more steps, each advantageously incorporating an apparatus in which the slurry is subjected to a continuous series of impacts and is chiefly in such a loading state in which the slurry can be thrown in a free space
30 in the inside of the mill. In the first slurring step, a greater portion of the solids is slurried in the water by dosing the solids either into the entering water or the circulating slurry. The latter approach is technically superior though expensive to implement. In the second
35 step, more solids is slurried into the slurry prepared in the first step.

Even more advantageously, the apparatus incorporates a pin mill or similar equipment in which dispersion in water is replaced by dispersion in air, whereby agglomerations and lumps are broken into a steady flow. Then, the solids discharged from the outlet of the disintegrating equipment are dosed directly via, e.g., a hopper feed pipe into the gap of the dispersing mill.

More specifically, the method according to the invention is characterized by what is stated in the characterizing part of claim 1.

Furthermore, the apparatus according to the invention is characterized by what is stated in the characterizing part of claim 11.

Other characterizing and preferred embodiments are disclosed in the appended dependent claims.

The invention offers significant benefits.

Solids can be dispersed to form a slurry in water or other liquid under such conditions in which lumps typical of the slurring process are prevented from forming putty-like, high-solids agglomerations with water. Additionally, such conditions can be created in which such putty-like agglomerations are disintegrated optimally. Thus, the invention makes it possible to disintegrate difficult-to-handle putty-like agglomerations with the help of a high-consistency slurry complemented with preceding disintegration of pigment solids in air prior to the slurring step.

The above-described approach also offers an improved solution to the dosing problem. When the slurry is transferred from the first step to either the second step or an intermediate storage, its density can be easily deter-

mined by means of, e.g., a continuously operating density gauge which directly indicates the dry solids. However, in the second step, where only a 20 - 30 % amount of the pigment is added remaining from the total amount of solids to be slurried, the viscosity is increased to multiple value relative to that of the first step.

In the following the invention will be examined in greater detail with reference to the appended drawings in which

Figure 1 is a diagrammatic illustration of an embodiment of the slurrying method according to the invention;

Figure 2 is a diagrammatic illustration of another embodiment of the slurrying method according to the invention;

Figure 3 is a diagrammatic illustration of a third embodiment of the slurrying method according to the invention;

Figure 4 shows an embodiment of the apparatus according to the invention; and

Figure 5 shows another embodiment of the apparatus according to the invention.

Referring to Fig. 1, the method illustrated therein implements the two-step mixing by means of a single mixer 1. The two-step mixing action in this arrangement is implemented by virtue of a return circuit 2. Into the mixer 1 is fed high-viscosity product taken from the product discharge line 5 of the mixer via the return circuit 2, whereby water is simultaneously added from line 4 and more solids from line 3. As the solids content of the slurry taken from the product discharge line 5 via

the return circuit 2 corresponds to the solids content of the final product, the return flow is diluted with water from the line 4 prior to its feed back into the mixer thus facilitating the mixing of more solids into it.

5 Accordingly, the mixer is utilized for effective two-step mixing. In the method shown in Fig. 2, the mixing is implemented using two mixers 1a and 1b in a manner which is easier to control. Into the mixer 1a are fed water and solids via lines 3 and 4, respectively, in such a ratio
10 that the solids content of the slurry discharged from the mixer 1a along a connecting line 6 is lower than that of the desired final solids content. At this point the slurry contains the final amount of liquid. In the second mixer 1b, the remaining amount of solids is mixed in the
15 slurry and the final product is discharged from the mixer along a product outlet line 5. The method shown in Fig. 3 is otherwise identical to that illustrated in Fig. 2 except that, besides the feed of solids, additional water is fed into the second mixer 1b via line 8. A benefit of
20 this arrangement is that the qualities of the slurry and particularly its solids content can be controlled in a more flexible manner.

Referring to Figs. 4 and 5, two apparatuses suited for
25 implementing the invention are shown. The reference numerals denoting the elements of the apparatus in these drawings are for equivalent parts the same as those used in Fig. 2. The two mixers 1a and 1b of the apparatus are adapted on a framework 11, and the mixers are driven by
30 means of electric motors mounted on the framework 11. The mixers 1a and 1b are located on the framework 11 so that the mixer 1a of the first mixing step is situated above the mixer 1b of the second mixing step. To the infeed nozzle of the first mixer 1a is connected a water line 4 and a solids feed arrangement 3. The solids feed arrange-
35 ment 3 comprises a feed hopper 9 and an auger feeder 10 driven by an electric motor. From the discharge nozzle of

the first mixer 1a passes a connection line 6 to the inlet nozzle of the second mixer 1b, and to the connection line is attached a second feed arrangement 7 of dry solids, similar to the feed arrangement of the first
5 mixing step. Starting from the discharge nozzle of the second step mixer 1b is connected a product discharge line 5 along which the prepared slurry is taken to a storage tank 13. To the bottom of the storage tank 13 is adapted a slurry outlet nozzle with an auger feeder 14
10 for pumping the slurry to the point of use.

Referring to Fig. 5, the apparatus shown therein is otherwise similar to that of Fig. 4 except that in the latter embodiment the first dry solids feed arrangement 3
15 is complemented with a mixer 15 suited for homogenizing the consistency of the dry solids through grinding the solids into a pulverized form. This approach prevents solids lumps from getting into the water and makes mixing easier. The homogenization of solids particle size is
20 advantageously carried out using a pin-mill-type mixer similar to that used for slurring, and in fact, such a mixer may also be connected to the solids feed arrangement 7 of the second mixing step. Besides different types of mixers, the particle size of the solids can be homo-
25 genized using various screening equipment. Homogenization of dry solids is particularly important when hygroscopic materials are mixed that easily agglomerate into lumps under humidity.

30 In addition to those described above, the invention may be implemented using a plurality of other kinds of apparatuses. When required, the number of the mixers may be increased. As described for the embodiment shown in Fig. 3, the second mixer is advantageously complemented
35 with a water feed connection, whereby the consistency of the slurry may be controlled as required by adding water. The slurring operation is implemented using different

variants of pin-mill-type mixers having at least two concentric rings carrying vanes and pins that impose large shearing forces on the material to be mixed. A portion of the rings with vanes may comprise nonrotating stator
5 rings, or alternatively, the perimeters can be arranged to rotate at different speeds in the same direction or in opposite directions.

The proportional amounts of dry solids to be fed in the
10 different mixing steps are determined by the properties of the materials to be mixed. For instance, several materials used for coating a paper web behave during mixing so that when the solids content of the slurry exceeds a certain limit, the viscosity of the slurry suddenly jumps
15 drastically. Obviously, such anomalies must be taken into account in the preparation of a slurry. Some materials are optimally mixed by a method in which the solids are advantageously mixed in either mixing step divided into half and half of the total dry solids to be
20 mixed. However, the amount of solids feed in the first mixing step should not be selected smaller than 50 % of the total solids, because otherwise the viscosity and density of the slurry remain insufficiently small and mixing in the second step may become difficult. In a
25 plurality of cases, the amount of solids to be mixed in the second step should preferably be less than 30 % of the total solids to be mixed. The present method is also suited for preparing other slurries than water slurries alone.

30

Example 1

In the example a slurry was prepared of calcined kaolin marketed under the tradename Norcal. In the first step,
35 the kaolin was slurried using an amount that made the solids content of the slurry to be 40 %. Slurrying was carried out in a pin mill having the pins replaced by

radially aligned impact stop vanes having a radial length of 4 cm. The mill had a total number of 5 concentric rings, of which 3 were rotor rings and 2 stator rings. The rotational speed was 1500 r/min and the outer diameter of the outermost ring was 450 mm. In such a mill the slurring action was achieved using one through-pass with a retention delay in the order of 0.1 s. After slurring the product was passed into a mixing tank where its consistency was homogenized as it was difficult to perform continuous dosing accurately at said extremely short mixing delay. Power input to the mill of the first mixing step was 5.5 kW.

The second mixing step was carried out using a mixing apparatus having two rotors rotating in opposite directions and in which the outer diameter of the outer rotor was 350 mm and the outer diameter of the other rotor was 300 mm. Both of the rotors had 3 rings. Besides the added kaolin, 0.5 % alkali (NaOH) and 0.12 % polyacrylate (Fennodisp) was added. The outer rotor of the second step mixer was run at +1500 r/min while the other rotor was run at -2200 r/min.

Kaolin was fed in both the first and the second step by a total amount corresponding to a slurry preparation capacity of 1200 kg/h dry kaolin. The input power to the rotor motors of both mixers was 5.5 kW and 5.5 kW, which corresponds to a specific energy consumption of 3.8 kWh per ton mixed dry solids.

In this example the homogenization of the dry kaolin was carried out prior to slurring using a screen. Screening was used for reasons of simplicity.

The final product was a slurry having a viscosity of 80 cP measured in the Brookfield viscometer using rotor no. 3 and having a solids content of 54.4 %.

Example 2

In this example a slurry was prepared by virtue of the method illustrated in Fig. 1 using a single-mixer apparatus operated with the same rotors as in the first example, and similarly, with the same input power to both rotor motors at 5.5 kW and 5.5 kW. The slurry was prepared by mixing conventional kaolin (trademark Komolco) in water to form a slurry having a final solids content of 74 %. The preparation of the slurry was carried out by branching half of the slurry flow discharged from the mixer aside into a sigmoidal hose pump which returned the recycled half back to the inlet nozzle of the mixer. Simultaneously, water and dry solids were fed into the mixer inlet in a ratio corresponding to final solids content of the slurry discharged from the mixer. The specific energy consumption was 7.6 kWh per ton mixed dry solids. The quality of the final product was normal and its screen residue using a 325-mesh screen was 0.1 %.

Claims:

1. A method of preparing a slurry from a liquid and dry solids by mixing the liquid and the dry solids in a mixer
5 (1) in which the substances to be mixed are passed through the mixer (1) as a continuous flow forming a slurry, c h a r a c t e r i z e d in that
- at least a portion of the total solids is mixed in
10 a predispersed slurry in such a quantity that the amount of added solids brings the solids content of the prepared slurry to desired level of the final product, and
 - the mixer (1) used in the method is an apparatus
15 having at least one concentric pair of rings and the rings are equipped with impact stops rotated so as to make the rotational speed difference between rings to be 20 - 200 m/s.
- 20
2. A method as defined in claim 1, c h a r a c t e r - i z e d in that said mixing is performed in two steps in a single mixer (1) so that at least a portion of the product flow exiting the mixer (1) is branched aside and
25 this portion is returned back in conjunction with the feed of said liquid and said solids into the inlet of said mixer.
3. A method as defined in claim 1 or 2, c h a r a c -
30 t e r i z e d in that the composition of said solids is homogenized with the help of, e.g., a screen or mixer just prior to contacting the solids with said liquid.
4. A method as defined in claim 1 or 3, c h a r a c -
35 t e r i z e d in that said mixing is performed in two steps using two separate mixers (1a, 1b) so that a portion of the total solids to be mixed is mixed with

said liquid in the first step, while the rest of the solids is mixed in the second step.

5 5. A method as defined in claim 4, c h a r a c t e r -
i z e d in that also more liquid is added into said
mixer (1b) in the second step.

10 6. A method as defined in any foregoing claim,
c h a r a c t e r i z e d in that at least half of the
total solids to be mixed is mixed with said liquid in the
first mixing step.

15 7. A method as defined in any foregoing claim,
c h a r a c t e r i z e d in that less than 30 % of the
total solids to be mixed is mixed with said liquid in the
second mixing step.

20 8. A method as defined in any foregoing claim,
c h a r a c t e r i z e d in that the mixer (1) used in
the method is a pin-mill-type mixer incorporating impact
stops mounted on concentric rotors so that at least a
portion of said impact stops are rotatable.

25 9. A method as defined in any foregoing claim,
c h a r a c t e r i z e d in that the mixer used in the
method is a pin-mill-type mixer incorporating impact
stops mounted on concentric rotors so that at least a
portion of said impact stops are rotatable and that in
the mixer (1) used for slurring said liquid with said
30 solids the speed difference between the concentrically
rotating impact stops is at least 10 m/s and that in the
mixer (15) used for homogenizing the composition of said
solids the speed difference between the concentrically
rotating impact stops is at least 20 m/s.

35

10. A method as defined in any foregoing claim,
c h a r a c t e r i z e d in that the mixer used in the

method incorporates concentric ring-like rotors displaced so that the mutual distance between the rotors is smaller than the radial distance between the inner and outer diameters of each rotor ring.

5

11. An apparatus for mixing a liquid and dry solids into a slurry, said apparatus comprising at least one mixer (1) incorporating at least two concentric rotor rings with impact stops mounted thereon and at least one of the rotor rings being rotatable, c h a r a c t e r i z e d
10 by

- means (2) for branching at least a portion of the slurry discharged from the mixer (1) to a second mixing step, and
- 15 - means (3) for adding solids into the slurry flow passed from the mixer to said second mixing step.

12. An apparatus as defined in claim 11, c h a r a c -
t e r i z e d by means (4) for adding liquid to said
20 second mixing step.

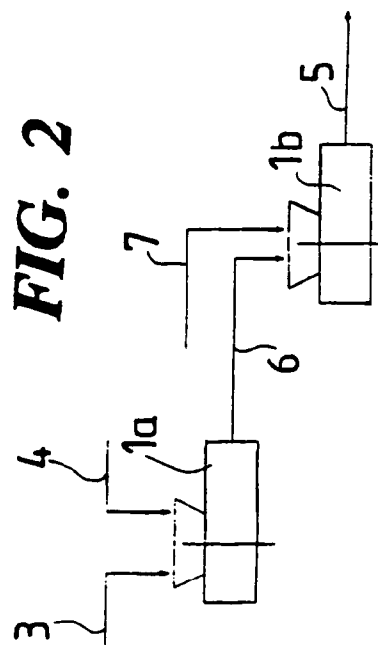
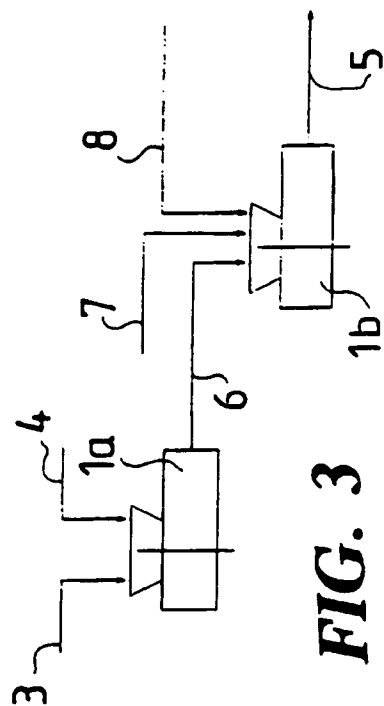
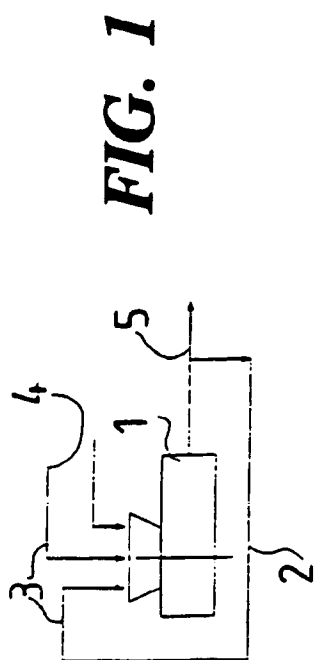
13. An apparatus as defined in claim 11 or 12,
c h a r a c t e r i z e d in that said apparatus in-
corporates a single mixer (1) and that said means for
25 passing the slurry discharged from said mixer (1) to said second mixing step comprise a return line (2) along which a portion of the slurry discharged from said mixer (1) can be recycled back to said mixer (1).

30 14. An apparatus as defined in claim 11 or 12,
c h a r a c t e r i z e d in that said apparatus incorporates two mixers (1) and that said means for passing the slurry discharged from the first mixer (1a) to the second mixer (1b) comprise a connection line (6)
35 along which the slurry discharged from the first mixer (1a) can be passed to the second mixer (1b).

15. An apparatus as defined in any of claims 11 - 14,
c h a r a c t e r i z e d by at least one pin-mill-type
mixer (15) suited for homogenizing the composition of
said solids prior to contacting said solids with said
5 liquid.

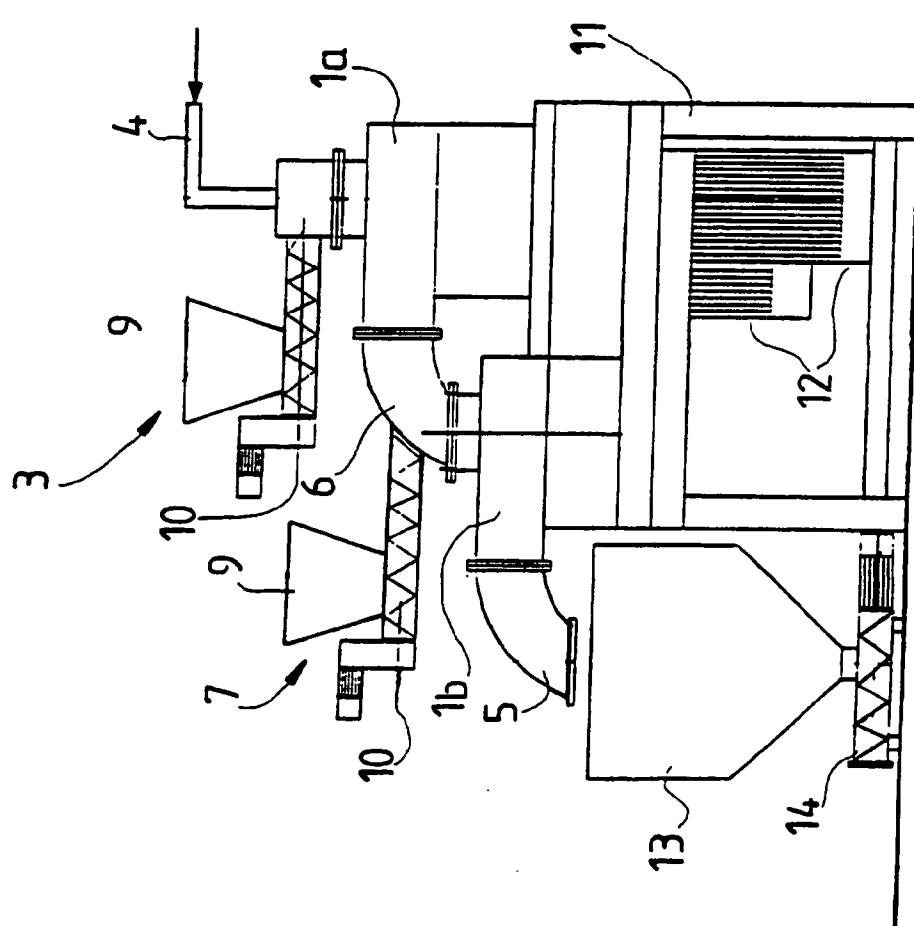
16. An apparatus as defined in any of claims 11 - 15,
c h a r a c t e r i z e d in that said mixers are pin-
mill-type mixers incorporating impact stops mounted on
10 concentric rotors so that at least a portion of said
impact stops are rotatable and that in the mixer (1) used
for slurring said liquid with said solids the speed
difference between the concentrically rotating impact
stops is at least 10 m/s and that in the mixer (15) used
15 for homogenizing the composition of said solids the speed
difference between the concentrically rotating impact
stops is at least 20 m/s.

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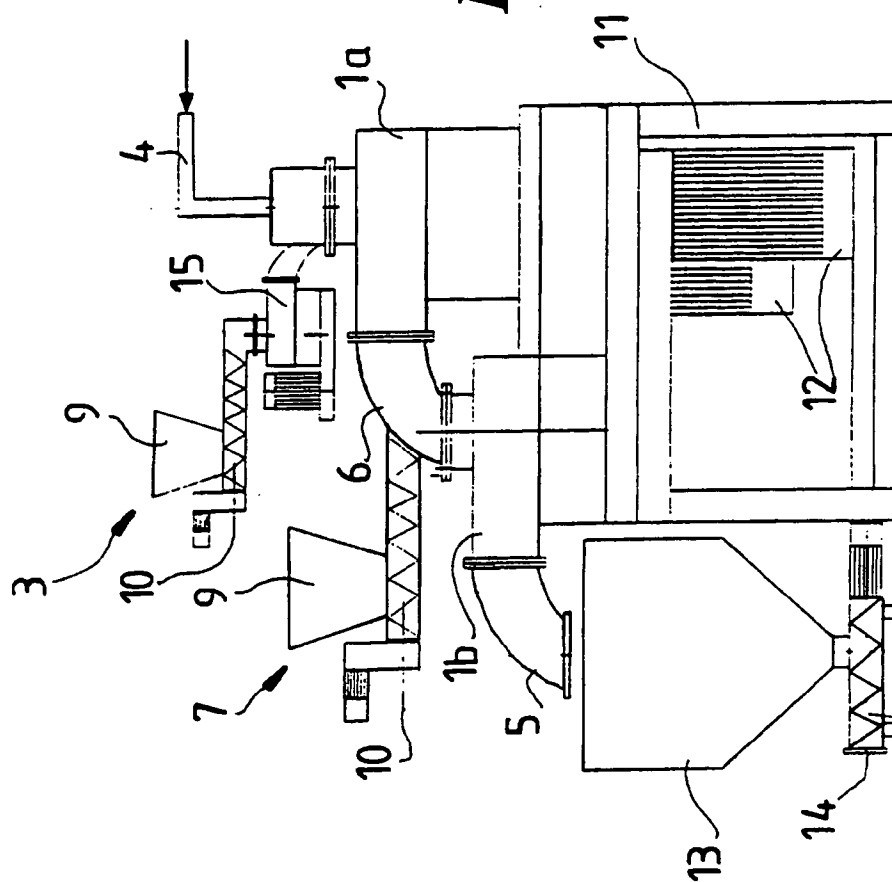


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FIG. 4



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FIG. 5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/FI 95/00675

A. CLASSIFICATION OF SUBJECT MATTER

IPC6: B01F 3/12

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC6: B01F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

DIALOG

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	FI 83897 B (FINNMINERALS OY), 31 May 1991 (31.05.91), claims 1-4	1
A	--	2-7
Y	EP 0079300 A1 (WATER-LINE S.A.), 18 May 1983 (18.05.83), figures 1-3, claim 1	1
A	-- -----	8-10, 15, 16

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:

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- * "P" document published prior to the international filing date but later than the priority date claimed

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

Information on patent family members

01/04/96

International application No.

PCT/FI 95/00675

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
FI-B- 83897	31/05/91	NONE	
EP-A1- 0079300	18/05/83	SE-T3- 0079300 CH-A,B- 649476 JP-C- 1660213 JP-B- 3027258 JP-A- 58081434 US-A- 4439042	31/05/85 21/04/92 15/04/91 16/05/83 27/03/84